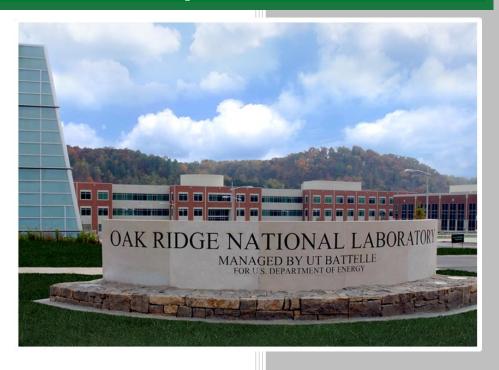
Assembly and Delivery of Rabbit Capsules for Irradiation of Reinforced Radiation Resistant SiC-SiC Composites in the High Flux Isotope Reactor



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Annabelle G. Le Coq Kory D. Linton Ryan C. Gallagher Yutai Katoh

July 23, 2018

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Materials Science and Technology Division

Assembly and Delivery of Rabbit Capsules for Irradiation of Reinforced Radiation Resistant SiC-SiC Composites in the High Flux Isotope Reactor

Annabelle G. Le Coq Kory D. Linton Ryan C. Gallagher Yutai Katoh

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ABSTRACT

The development of reinforced radiation resistant composites presents a critical challenge for invessel component application in advanced high temperature reactors. In its pursuit of this, Physical Optics Corporation (POC) is interested in developing a silicon carbide (SiC)-based ceramic matrix composite (CMC) with improved thermal conductivity in a high neutron radiation environment. The US Department of Energy (DOE) is supporting research efforts to develop Advanced Reactor Technology. Ultimately, the results of this project will determine the viability of using SiC matrix composites in hot structures in advanced high temperature reactors. The first objective of this project is to irradiate torsion and bend bar specimens in the High Flux Isotope Reactor (HFIR) and develop an initial data set to evaluate the evolution of composite mechanical strength, dimensional changes, and thermal conductivity. This report briefly describes the irradiation experiment design concepts, summarizes the irradiation test matrix, and reports on the successful delivery of three rabbit capsules to the HFIR. Rabbits of both torsion and bend bar configurations have been assembled, welded, evaluated, and delivered to the HFIR along with a complete quality assurance fabrication package.

1. INTRODUCTION

The high-temperature strength, oxidation resistance, and irradiation stability of silicon carbide (SiC) are properties which have led researchers to explore SiC as a candidate material for a variety of nuclear applications [1-6]. However, in a harsh advanced reactor environment, with high neutron radiation and high temperature, SiC thermal conductivity degrades resulting in a large temperature gradient through the composite. These large temperature gradients create a complex stress state due to differential swelling and could damage SiC components [7]. Thus, mechanical testing of irradiated SiC materials is essential for irradiation effect investigations The US Department of Energy (DOE) awarded Physical Optics Corporation (POC) a grant to experimentally investigate irradiation effects in a SiC-based ceramic matrix composite (CMC), with an improved thermal conductivity. The results obtained from this irradiation testing will provide an initial data set to evaluate the evolution of the composite properties subjected to a high neutron flux.

Irradiation capsules have been designed and fabricated to allow PyC coated SiC specimens to be irradiated in the core of the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). Post-irradiation examination of the irradiated specimens will include mechanical strength analysis, dimensional changes, and thermal conductivity evolution. This work summarizes the assembly and delivery of three rabbits containing SiC CMC specimens to the HFIR. The specimens include rectangular bend bar specimens for flexural testing and hourglass specimens for torsion testing. This report provides a brief overview of the irradiation test matrix, the capsule design concepts, and the successful delivery of all irradiation capsules to the HFIR.

2. EXPERIMENT DESIGNS AND TEST MATRIX

2.1 BEND BAR CAPSULE DESIGN

The bend bar irradiation capsule design is shown in Figure 1. This design places three bend bar specimens in a V-4Cr-4Ti holder at the center of the rabbit housing. The specimens are surrounded by chemical vapor deposited (CVD) SiC blocks, Inconel springs, and SiC liners, to maintain the specimens in place, allow them to swell under irradiation, and prevent their contact with the holder. Two silicon carbide passive temperature monitors are placed on the outside of the CVD SiC blocks for post irradiation dilatometry. The holder is positioned in an aluminum housing, which is directly cooled by the reactor primary coolant. The nominal bend bar temperatures were determined to be around 650°C by scaling the gamma heating rate of a previously demonstrated design, meant for 800°C with respect to a new position in the HFIR. The temperature will be validated using the SiC temperature monitors.

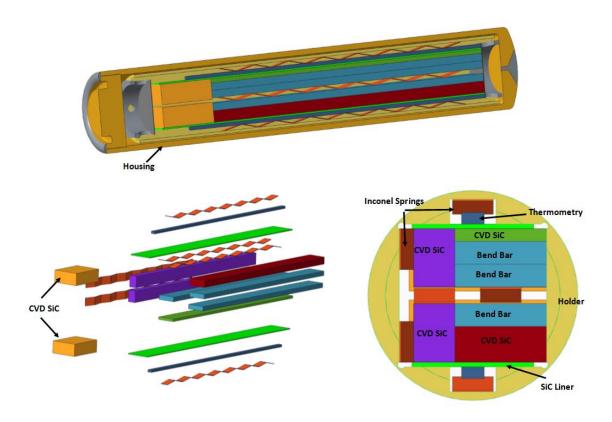


Figure 1. Capsule design concept for irradiating SiC matrix composite bend bars specimens.

2.2 TORSION CAPSULE DESIGN

Figure 2 shows the concept for the torsion rabbit design. Two rows of eight hourglass specimens stacked in the vertical direction are set inside a molybdenum holder. A center spacer separates the two rows of specimens while two retainer springs maintain the specimens centered in their holder. Molybdenum wires are used to secure the specimen stack within the holder. Two silicon carbide passive temperature monitors are placed on the outside of the CVD SiC blocks for post irradiation dilatometry. Grafoil insulator disks as well as quartz wool are placed at the top and bottom of the housing assembly reduce axial heat losses from the top and bottom hourglass specimens to the cooler surface of the capsule housing. The design was based on, and nearly identical to, a previous irradiation experiment. Finite element modeling of the previous design predicts 800°C average specimen temperatures. The thermal performance will be validated by the SiC temperature monitors, post-irradiation. Figure 3 displays the temperature contour plot for the torsional specimens.

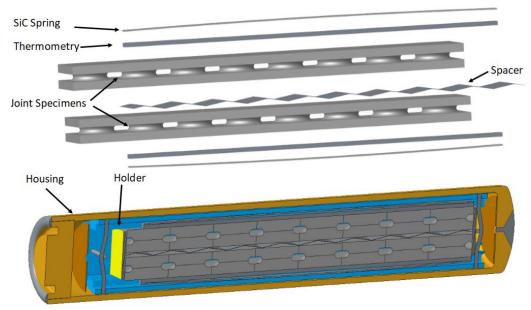


Figure 2. Capsule design concept for irradiating SiC matrix composite hourglass specimens.

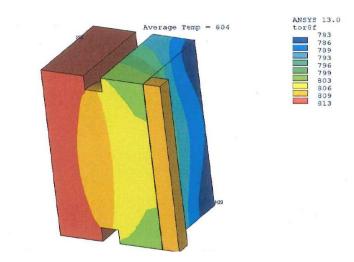


Figure 3. SiC matrix composite hourglass specimen temperature (°C) contours during irradiation.

2.3 IRRADIATION TEST MATRIX

Table 1 summarizes the irradiation test matrix. The specimens have been manufactured by POC. Each specimen was coated with PyC. Table 1 shows the loading of specimens in each rabbit along with the irradiation positions and fill gases. Each bend bar rabbit contains three bend bars while the torsion rabbit contains sixteen specimens. One bend bar rabbit will be irradiated for one cycle (cycle 481) in the HFIR, which will result in a radiation dose of approximately 1.85 dpa. The other bend bar rabbit and the torsion rabbit will be irradiated for two cycles (cycles 481 and 482) in the HFIR, which will result in a radiation dose of approximately 3.7 dpa. The targeted specimen surface temperature is approximately 650°C for the bend bars and 800°C for the joint specimens. Temperature gradients through the thickness depend on the heat flux and the specimen thermal conductivity, which varies with specimen type and neutron fluence. The nominal bend bar specimen dimensions are 42 mm length, 4 mm width, and 1 mm thickness; the nominal hourglass specimen dimensions are 6 mm × 6 mm square section and 2.8 mm height.

Table 1. Rabbit irradiation test matrix showing the loading of specimens within each rabbit, the irradiation positions, and fill gases

Rabbit	Number of irradiation cycles	Specimens	Irradiation position	Fill gas
DCM01	1	1BB1, 1BB3, 2BB7	ТН-6	Ne*
DCM02	2	1BB2, 1BB6, 2BB9	TH-6	Ne*
DCM03	2	6B01, 6B02, 6B03, 6B04, 6B05, 6B06, 6B07, 6B08, 6C01, 6C02, 6C03, 6D01, 6D02, 6D04, 6D05, 6D06	PTP-2	Ar

^{*} Or Ne equivalent

3. RABBIT CAPSULE ASSEMBLY AND DELIVERY TO THE HFIR

3.1 BEND BAR RABBITS ASSEMBLY

The two bend bar rabbits (DCM01 and DCM02) were assembled. Pictures of the complete parts layout and the parts in the holder sub-assembly for rabbit DCM01 are shown in Figure 4. Figure 5 shows the parts layout for rabbit DCM02. The signed capsule fabrication request forms are provided in APPENDIX A. Figure 6 shows a top-down view of SiC matrix composite bend bar specimens assembled inside the rabbit housing, with liners, springs and thermometry.

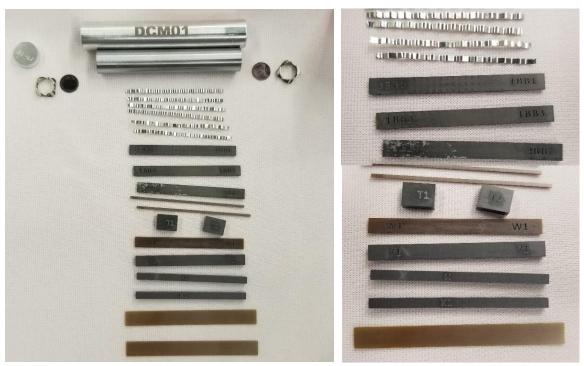


Figure 4. Parts (left) and sub-assembly components (right) of rabbit DCM01.



Figure 5. Parts (left) and sub-assembly components (right) of rabbits DCM02.

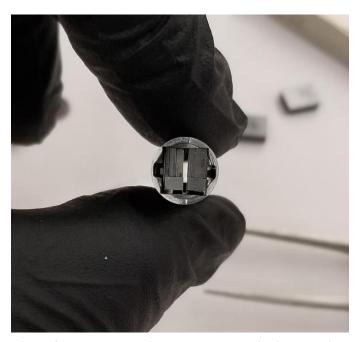


Figure 6. Top-down view of bend bar specimens assembled inside rabbit housing DCM01.

All capsule components were dimensionally inspected and cleaned according to HFIR-approved procedures, drawings, and sketches. After assembly of the internal components, all rabbit housing end caps were welded to the housings using an electron beam weld. The capsules were then placed inside sealed chambers that were evacuated and backfilled with an 59.5% Ar–He balance mixture gas three times to ensure a pure environment. The chambers were placed inside a glove box, which was also evacuated and backfilled with 59.5% Ar–He balance mixture gas. Each rabbit had a small hole in the bottom of the housing that was sealed using a gas tungsten arc welding procedure. All welds passed visual examination. Each capsule was then sent for nondestructive examination, which included a helium leak test, hydrostatic compression at a pressure of 1,035 psi, mass comparisons before and after hydrostatic compression to ensure no water penetrated the capsule housing, and a final post-compression helium leak test. All rabbits passed helium leak testing and hydrostatic compression.

3.2 TORSION RABBIT ASSEMBLY

One torsion rabbit (DCM03) was assembled. A picture of the layout before irradiation is shown for this rabbit in Figure 7. The torsion rabbit and rabbit components were inspected, cleaned, assembled, and tested using the same processes and procedures as the bend bar rabbits, except that the backfill gas was pure argon instead of an 59.5% Ar–He balance mixture. All rabbits passed weld examination, leak testing, and hydrostatic compression.

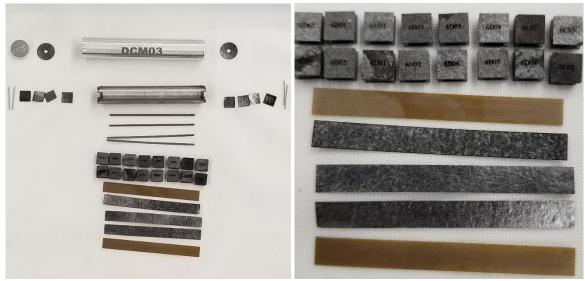


Figure 7. Capsule parts for torsion rabbit DCM03 (left), and the joint specimens with liners and spacers (right).

3.3 QUALITY ASSURANCE, FABRICATION PACKAGE, AND DELIVERY TO THE HFIR

Each rabbit irradiation experiment requires a fabrication package that is reviewed by an independent design engineer, a lead quality assurance (QA) representative, and a HFIR QA representative before acceptance for insertion into the HFIR. The fabrication package must satisfy the requirements of the Experiment Authorization Basis Document (EABD). Rabbit capsules fall under document EABD-HFIR-2009-004. This document specifies a number of requirements that the rabbits must satisfy in the areas of

- thermal safety analyses,
- material certification,
- dimensional inspection,
- cleaning,
- assembly procedure,
- sample loading,
- fill gas,
- welding, and
- nondestructive evaluation.

A unique fabrication package was prepared for the bend bar and torsion rabbit capsules. This package was reviewed and approved by an independent design engineer, lead QA representative, and HFIR QA representative and accepted by HFIR on July 12, 2018. The final signed acceptance page of the EABD is provided in APPENDIX A. All three rabbits are scheduled for insertion during HFIR cycle 481 (July 2018).

4. SUMMARY AND CONCLUSIONS

This work summarizes the capsule design concepts and the irradiation test matrix for three rabbit capsules, which were successfully assembled and delivered to the HFIR for insertion during cycle 481 (July 2018). Each bend bar rabbit contains three SiC matrix composite bend bars; the torsion rabbit contains sixteen SiC matrix composite hourglass specimens. The specimens will be evaluated post-irradiation to investigate the effects of irradiation on the composite mechanical strength, dimensional changes, and thermal conductivity. The rabbits were successfully assembled, welded, evaluated, and delivered to the HFIR along with a complete QA fabrication package. Pictures of the rabbit assembly process are included in this report. Documentation of the capsule fabrication and final acceptance by HFIR is provided in an appendix. Ultimately, the results of this project will determine the viability of using silicon carbide matrix composites in hot structures in advanced high temperature reactors.

5. WORKS CITED

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APPENDIX A. FABRICATION AND QUALITY ASSURANCE DOCUMENTATION FOR COMPLETED RABBITS

APPENDIX A. FABRICATION AND QUALITY ASSURANCE DOCUMENTATION FOR COMPLETED RABBITS

0.51677 0.50962 0.50270 0.0660

0.0670 0.4070 0.4060

0.6220 0.6220 0.3170 0.0320 0.0640

Comment

Rev.

Drawing S18-10-BENDBAR X3E020977A633 59.5% Argon/Bal He

> Assembly Drawing Welding & Cleaning Fill Gas

Assembly

0.8380

5/2/2018

Capsule Fabrication Request Sheet

Capsule Number:	DCM01	١.				Approvals				
Irradiation Conditions							Request		Build	
Irradiation Location	푸		9			Performed by:	8 Sh	5/8/20(6	Sec. By	13
First Cycle Goal Irradiation Time Irradiation Temperature		10.	480 1.0 cycles 650 °C			Checked by:	Ž	2/8/10	B- 2h	LO.
Housing Fill Gas		98	59.5Ar/He							
Capsule Fabrication	Denvisor	3	1	Motorial	2	1	2	0.00	5	2
Housing	X3E020977A634	Y Y	rarı	AL 6061	Count	Comment	20713	20713	17-113	≅ 4
End Cap	X3E020977A634	4	2	AL 4047	-		20714	20714	17-78	10
KAPL Bend Bar Holder	X3E020977A325	0	9	V-4Cr4Ti	-		18790	20795	1-15-05-04	~
Centering Thimble	X3E020977A540	0	τ-	304 SS	2	51, 52	19583	19583	2 Totai	0
KAPL Bend Bar Spring	X3E020977A325	0	80	Inconei	8		20796	20796	6 Total	0
Bend Bar Specimen	S18-11-BENDBAR	0	-	SiC	60		20797	20797	1881	O,
									1BB3	Ö
									2887	이
Passive Thermometry	X3E020977A325	0	10	Sic	2		19502	20708	329	0
							2002	20130	380	
Specimen	S18-11-BENDBAR	_	v.	SIC	2	78×6×979	20883	20867	Ţ	0
							2002	2004	T2	0
Specimen	S18-11-BENDBAR	0	က	SiC	-	42 x 4 x 0.55	20863	20865	W1	0
Specimen	S18-11-BENDBAR	0	4	SiC		42 x 4 x 1.56	20863	20866	۸,	0
Specimen	S18-11-BENDBAR	0	2	SiC	64	42 x 2 55 x 1 8	20863	20864	×	0
						200000000000000000000000000000000000000	2007	2	X1	0
Liner	S12-13-MC02	0	v	SiC	2	3A, 4A	19502	20028	2 Totaí	0
Quartz Wool	S18-10-BENDBAR	0	က	Si02		As Needed	20679	20679		0
Support disk	S18-10-BENDBAR	0	2	Moly	2		19593	19593	2 Total	0
								100	Specimen Mass	Π.
									Internal Mass	
Accomply										

Capsule Fabrication Request Sheet

Capsule Number:	DCM02	Approvals	
Irradiation Conditions			Request
Irradiation Location	TH	Performed by:	A. 2h 51820
First Cycle Goal	480	Checked by:	7
Irradiation Time	2.0 cycles		18/2 X X X
Irradiation Temperature	C 850 °C		
Housing Fill Gas	59.5Ar/He		

5/2/2018

Build

Capsule Fabrication										
	Drawing	Rev.	Part	Material	Count	Comment	MATIR	FAB IR	QI	Mass (g)
Housing	X3E020977A634	٩	-	AL 6061	-		20713	20713	17-116	4.3123
End Cap	X3E020977A634	٨	2	AL 4047	-		20714	20714	17-79	0.5154
KAPL Bend Bar Holder	X3E020977A325	0	ဖ	V-4Cr4Ti	-		18790	20795	1-15-05-09	7.1020
Centering Thimble	X3E020977A540	0	-	304 SS	2	51, 52	19583	19583	2 Total	0.4200
KAPL Bend Bar Spring	X3E020977A325	0	80	Inconel	9		20796	20796	6 Total	0.2480
Bend Bar Specimen	S18-11-BENDBAR	0	-	Sic	က		20797	20797	1882	0.51420
									1886	0.51850
									2889	0.49570
Passive Thermometry	X3E020977A325	0	9	SiC	2		10507	20709	308	0.0860
					L		20061	20102	377	0.0870
Specimen	S18-11-BENDBAR	C	u	SiC	2	78 48 4 7 7 7	20,883	20867	T3	0.4070
		>	>			21.24040.7	20002	70007	14	0.4060
Specimen	S18-11-BENDBAR	0	က	SiC	-	42 x 4 x 0.55	20863	20865	W2	0.2740
Specimen	S18-11-BENDBAR	0	4	SiC	+	42×4×1.56	20863	20866	72	0.8380
Specimen	S18-11-BENDBAR	0	2	SiC	2	0 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7	00000	V 2000	X11	0.6220
						42 X 2.33 X 1.0	Znogo	20004	X111	0.6220
Liner	S12-13-MC02	0	-	SiC	2	3A, 4A	19502	20028	2 Total	0.3170
Quartz Wool	S18-10-BENDBAR	0	ဗ	SiO2		As Needed	20679	20679		0.0320
Support disk	S18-10-BENDBAR	0	2	Moly	2		19593	19593	2 Total	0.0640
									Total Mass	17.8411
									Specimen Mass	4.6974
								_	Internal Mass	13,0134

Assembly			
	Drawing	Rev.	Comment
Assembly Drawing	S18-10-BENDBAR	0	
Welding & Cleaning	X3E020977A633	0	
Eill Gee	50 500 Amon/Rol Ho		

Page 1 of 1 tte 5/10/2018

Capsule Fabrication Request Sheet

	•			Date 6/10/2018
Capsule Number:	DCM03	Approvals		
Irradiation Conditions			Request	Build
Irradiation Location	PTP 2	Performed by:	8-24 SEIZO18	wallen Stolks
First Cycle Goal	480	Checked by:	8	4 94
Irradiation Time	2.0 cycles		\$/10/18	5/2/2018
Irradiation Temperature	S00 €		21.1.1	
Housing Fill Gas	Argen			

Capsule Fabrication

Capsule Fabrication										
	Drawing	Rev.	Part	Material	Count	Comment	MATIR	FAB IR	₽	Mass (a)
Housing	X3E020977A634	⋖	-	AL 6061	-		20713	20713	17-117	4.3001
End Cap	X3E020977A634	4	2	AL 4047	-		20714	20714	17-75	0.5135
Specimen Holder	\$13-22-TOR03	2	2	Moly	-		20026	20234	M1003	14.8810
Holder Support Wire	\$13-22-TOR03	2	65	Moly	4		19600	19600	4 Total	0.1040
Specimen Support Foil	S13-22-TOR03	2	4	Moly	2		19593	19593	2 Total	0.0510
Small Thermometry	X3E020977A540	1	3	SiC	2	339,391	19502	19709	2 Total	0.1330
Retainer Spring	\$13-21-TOR02	0	2	SiC	2		20236	20236	2 Total	0.0450
Center Spacer	\$13-22-TOR03	2	7	grafoil	6		19812	19812	3 Total	0.1100
Joint Specimen	\$13-22-TOR03	7	ın	Sic	16		20797	20822	6801	0.187
									6802	0.181
									6089	0.185
									6804	0.176
									6805	0.192
									6B06	0.186
									6B07	0.186
									6B08	0.188
							,		6C01	0.172
									9002	0.175
									6003	0.179
									6D01	0.179
									6D02	0.172
		_							6D04	0.171
									6D05	0.174
									9009	0.178
Insulator Disk	S13-22-TOR03	2	6	grafoil	8		19812	19812	8 Total	0.0450
Liner	S12-13-MC02	0	-	Sic		Parts 1A & 2A	19502	20028	2 Total	0.3140
Quartz Wool	S13-22-TOR03	2	9	SiO2		As Needed	20679	20679		0.0310
								Ì		

Assembly			
	Drawing	Rev.	omment
Assembly Drawing	S13-22-TOR03	2	
Welding & Cleaning	X3E020977A633	0	
Fill Gas	Argon		

Experiment Authorization Bases Document: EABD-HFIR-2009-004

Date:01/12/2017

Title: Rabbit Irradiations in the HFIR Target Region Prepared By: G. J. Hirtz

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oz*li≥lz₀≀⊵* Date

Note: This section is used to documen	te of As-Built Experiment Capsule int acceptance of the as-built experiment for reactor in: after completion of Section 2. See notes for explanat	stallation and ion of
User I.D. PS HFIR I.D.*	d Heat Generation Classification (High or Low) Heat Classification ↓○ ພ	
Demot 11 - Mes LONG	10 W	
DCW09 ICW09	hish	
DONO3 DONOS	""	
	*	
	*	
* Quality Assurance to verify correlation presented on product body. Independent Verification of User I.D. at 2. Attach Capsule Fabrication Reques	A 911/1	with markings
	0 0	
3. Approvals (see notes for explanation Ryun Gullasher Lead Experimenter	Lead Experimenter (signature)	7/5/18 Date
Mark C. Vance Lead QA	Lead QA (signature)	7/9/18 Date
Brian L Wingfield RRD QA	RRD QA (signature)	740/18 Date
RRD EA&C Staff	RRD EA&C \$taff (signature)	Date Date
N.A. No NCS Requirements RRD Criticality Safety Officer	N.A. No NCS Requirements RRD Criticality Safety Officer (signature)	Date
N.A. No MBA Requirements HFIR MBA Representative	N.A. No MBA Requirements HFIR MBA Representative (signature)	Date

Fue Beาคม ซ์ HFIR Operations (print name)